Preliminary results of CoQtz-N: A quartz reference material for terrestrial in-situ cosmogenic ^{10}Be and ^{26}Al measurements

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Abstract

There is growing interest in geochronological applications of terrestrial *in situ*-produced cosmogenic nuclides, with the most commonly measured being ^{10}Be and ^{26}Al in quartz. To extract and then separate these radionuclides from quartz and prepare them in the oxide form suitable for accelerator mass spectrometry (AMS) requires extensive and careful laboratory processing. Here we discuss the suitability of a crushed, sieved and etched, sub-aerially exposed vein quartz specimen (CoQtz-N) to act as a reference material for chemical laboratory preparation and AMS measurements. Splits of CoQtz-N were distributed to eleven target preparation laboratories. The CoQtz-N ^{10}Be targets were then measured at seven different AMS facilities and five of the preparation laboratories had their ^{26}Al targets measured at four different AMS facilities. We show that CoQtz-N splits are sufficiently homogeneous with regard to nuclide concentrations, that it has been cleaned of any atmospheric derived (i.e. meteoric) ^{10}Be and that it has low concentrations of the major elements that can interfere with Be and Al extraction chemistry and AMS measurements. We derive preliminary concentrations for ^{10}Be and ^{26}Al in CoQtz-N as 2.53 ± 0.09 x10^6 at/g and 15.6 ± 1.6 x10^6 at/g, respectively, at the 95% confidence limit.

Keywords: ^{26}Al; ^{10}Be; CoQtz-N; Cosmogenic nuclides; Reference material.

1. Introduction

Over the last few decades there has been a growing interest in measuring cosmic ray-produced ^{10}Be and ^{26}Al within quartz to determine rates of geomorphological processes, surface exposure ages or burial histories (Granger et al., 2013). The trace amounts of ^{10}Be and ^{26}Al produced in quartz require sensitive measurements by accelerator mass spectrometry (AMS) and the preparation of geological samples into a suitable form for such analysis requires careful processing in the laboratory. Several different approaches for preparing ^{10}Be and ^{26}Al AMS targets from cleaned quartz separates are used by different laboratories (e.g. Binnie et al., 2015; Child et al., 2000; Corbett et al., 2016; Kohl and Nishiizumi, 1992; von Blanckenburg et al., 2004), while AMS measurements are calibrated against various standard reference materials and performed using different accelerator configurations and analysis schemes (Merchel et al., 2012). There is, therefore, a need to be able to compare the ^{10}Be and ^{26}Al concentrations (i.e. atoms/gram sample) being reported by different groups with respect to their chemistry processing techniques and their analytical performance.

The standard method of analyzing how well results from different laboratories agree is by inter-laboratory comparison, or proficiency testing. Such trials allow laboratories to examine their performance and make corrections where needed (Scott et al., 2007). However, this requires available common materials, ideally of known, or at least agreed-upon (i.e. nominal), values. Inter-laboratory comparisons were carried out as part of the Cronus-Earth and Cronus-EU projects (Phillips et al., 2016) using rock samples prepared from appropriate lithologies. This work investigated the suitability of specific materials for cosmogenic ^{21}Ne (Vermeesch et al., 2015), ^{3}He (Blard et al., 2015), ^{10}Be and ^{26}Al measurements (Jull et al., 2015). One bedrock and one beach sand specimen, termed CRONUS-A and CRONUS-N, respectively, were collected and measured by several groups participating in the Cronus projects for the purpose of comparing ^{10}Be and ^{26}Al. However, concerns have been raised regarding the

homogeneity of CRONUS-N (Bierman et al., 2017), while the consensus ^{10}Be concentration value derived for CRONUS-A is very high, at 3.303 x10^7 at/g (Jull et al., 2015). Therefore, there is still the need for quartz reference materials, with lower concentrations more typical of the majority of ^{10}Be and ^{26}Al measurements, which can be prepared and measured alongside samples. In addition, the Cronus results highlight a need for further comparison studies of ^{10}Be and ^{26}Al (Jull et al., 2015). Having a full procedural reference material that is pre-etched quartz as opposed to a calibration reference material in the form of Be oxide, for example (Merchel et al., 2012), allow comparisons to be made not only of the AMS but also the results from the sample preparation laboratories. However, this introduces the added complexity of determining whether scatter, or any bias observed, derives from preparation of samples as targets in the laboratory or during the AMS measurement.

In this contribution we present details of the preparation and the initial measurements of CoQtz-N, a quartz material that we recommend should be used as an in-house, chemistry procedure reference material by the AMS community, in proficiency and full reproducibility testing, as well as for collaborative inter-laboratory investigations. We discuss the intra- and inter-laboratory results and present preliminary consensus values for the concentrations of ^{10}Be and ^{26}Al. Finally, we briefly discuss potential reasons for the differences observed in results from different groups and consider what remains to be investigated to mitigate these differences.

2. CoQtz-N production, material tests and concentration determinations

2.1. CoQtz-N production and distribution

The CoQtz-N quartz material was produced from a single boulder of vein quartz that was part of a lag deposit mantling a low relief surface near Hakos in Namibia (Figure 1). The sample was broken-up at the University of Cologne with a sledgehammer and fragments were visually inspected for any impurities; a

few fragments that contained black tourmaline were discarded. The quartz was then crushed with a jaw crusher and disc mill and dry sieved to obtain the 250 – 710 µm size fraction. This portion was sent to the University of Edinburgh where it was etched three times in an ultrasonic bath using a 2% HF / 2% HNO_3 acid solution with a ratio of 1 g of quartz per 20 ml of dilute acid mixture. The final etch used analytical grade acids diluted in 18 MΩ·cm deionized water. The total quartz mass lost due to this etching was ~15%. Around 25 kg of etched quartz was returned to the University of Cologne where it was homogenized and then split using an 8-way rotating cone splitter fed by a vibratory chute. Splits of ~165 g were packaged in screw-top plastic beakers, labeled and distributed between 2014 and 2016 to 11 cosmogenic nuclide sample preparation laboratories, herein termed 'laboratories' for brevity (Table 1). CoQtz-N recipients were instructed not to clean the quartz any further but to dissolve it directly from the container. Five grams was suggested to be an appropriate, though not obligatory amount to dissolve in order to mimic routine samples but not exhaust the supply of CoQtz-N too rapidly.

2.2. Material tests

The purity of CoQtz-N was determined by ICP-OES (inductively coupled plasma - optical emission spectrometry) measurements at the University of Cologne. In brief, five 1 g portions were dissolved in concentrated trace-metal grade HF/HNO_3, heated to dryness three times in the presence of aqua regia before each sample was prepared as four dilutions for standard addition in 0.3M HNO_3. One reagent blank was prepared the same way in parallel with the samples. Measurements were performed on a Spectro Arcos ICP-OES using an axial configuration.

Subsequent to receiving some initial results, it was found that one laboratory had acid etched a portion of their CoQtz-N samples prior to dissolution. To test that additional etching would not influence the nuclide concentrations, a batch of six CoQtz-N splits were prepared at the University of Cologne, where

two of the splits were dissolved directly from the container, i.e. no further etching, two of the splits were etched once and two splits were rinsed and etched a second time. Etching in these cases was overnight in an ultrasonic bath, using a 1% HF / 1% HNO_3 acid solution with a ratio of 1 g of quartz per 100 ml of dilute acid mixture Specified grade acids and 18 MΩ·cm water were used. There was an approximate 20% loss of quartz mass from the splits that were etched twice.

2.3. Concentration determinations

Eleven laboratories reported CoQtz-N measurements of ^{10}Be undertaken on seven different AMS facilities and five of those laboratories also prepared and measured ^{26}Al targets that were measured on four of the seven different AMS facilities (Table 1). Herein, these AMS facilities are labelled as AMS 1 through AMS 7. Reporting from each recipient was done via a standardized Microsoft Excel® spreadsheet that asked for the following information: the ^{10}Be/^{9}Be, or ^{26}Al/^{27}Al, ratio for each CoQtz-N sample and relevant reagent blank; the mass of ^{9}Be in the carrier added to each CoQtz-N sample and relevant reagent blank; the name/brand and concentration of Be and Al carriers used, plus the final mass of ^{27}Al in the samples and blanks and the method of this determination. For each measurement reported, calculations following Eq. 1a and Eq. 1b for the concentration of ^{10}Be (N_{Be}) and ^{26}Al (N_{Al}) in atoms per gram were performed by the spreadsheet.

$$N_{Be} = \frac{\left[\left(\frac{^{10}Be}{^{9}Be}\right)_{Q} \cdot c_{BeQ} \cdot L \cdot \frac{1}{u_{Be}}\right] - \left[\left(\frac{^{10}Be}{^{9}Be}\right)_{B} \cdot c_{BeB} \cdot L \cdot \frac{1}{u_{Be}}\right]}{m}$$

Eq. 1a

$$N_{Al} = \frac{\left[\left(\frac{^{26}Al}{^{27}Al}\right)_{Q} \cdot c_{AlT} \cdot L \cdot \frac{1}{u_{Al}}\right] - \left[\left(\frac{^{26}Al}{^{27}Al}\right)_{B} \cdot c_{AlB} \cdot L \cdot \frac{1}{u_{Al}}\right]}{m}$$

Eq. 1b

In equations [1a] and [1b], subscripts *Q* and *B* on the $^{10}Be/^{9}Be$, or $^{26}Al/^{27}Al$, ratios are the CoQtz-N sample and reagent blank AMS measurements, respectively. The mass of ^{9}Be carrier used in the sample and blank are given by c_{BeQ} or c_{BeB}, respectively. The total ^{27}Al mass in the sample is given by c_{AlT} and in the blank by c_{AlB}. Note, c_{AlT} refers to the ^{27}Al within the quartz plus any ^{27}Al added as carrier. *L* is Avogadro's constant, u_B and u_{Al} are the respective atomic masses of ^{9}Be and ^{27}Al. The mass of CoQtz-N dissolved is denoted by *m*. Laboratories were asked to give final AMS analytical measurement uncertainties and the stable isotope mass uncertainties as one standard deviation. The uncertainties in the concentrations of ^{10}Be and ^{26}Al were also calculated by the spreadsheet, using standard error propagation formula (Bevington and Robinson, 2003). Additionally, CoQtz-N recipients were asked: what metal matrix was mixed with the BeO and Al_2O_3 before samples were pressed into targets and if this matched that in the AMS standard reference material (SRM) used for calibration; which AMS SRMs were used for the measurement; what nominal values were used for the AMS SRMs and in the case of $^{10}Be/^{9}Be$ standards, what value was assumed for the half-life of ^{10}Be.

3. Results and statistical analysis

3.1. Material purity

ICP-OES measurements of the concentrations of the major elements of most interest in CoQtz-N are given in Table 2. The concentration of Al is relatively low at 18.4 ± 0.6 ppm. Ti is sometimes cited as problematic for causing reduced Be chemistry yields and lower AMS ^{9}Be negative ion-source beam currents (e.g. Hunt et al 2008), but the Ti concentration is also relatively low at 7.6 ± 2.6 ppm. No differences in ^{10}Be concentrations were observed due to further etching of the CoQtz-N, as can be seen

by comparing the results of the etched and non-etched concentrations (Figure 2), so we do not exclude any results where additional HF etching was performed.

3.2. Initial measurements and corrections

Figure 3a shows the results from laboratories 1, 2, 3, 4 and 7 for the 63 BeO targets that were all measured on AMS 1, plotted against their average analyzed ^{9}Be current as a fraction of the average ^{9}Be current of the primary standards used in that run (relative ^{9}Be current). A significant positive correlation is observed with respect to ^{10}Be concentration versus relative ^{9}Be current, as well as a degree of clustering per laboratory. This explains an initially observed significant difference in the average ^{10}Be concentrations between laboratories 1, 2, 3 and 4 measured on AMS 1. Consequently, the ^{10}Be concentrations that were measured on AMS 1 were re-normalized to the average current of the standards used in the same run (Figure 3b). This produces an increase in the average ^{10}Be concentrations of laboratories 1, 2 and 4 of 0.8%, 5.1% and 5.3%, respectively, and means that the average ^{10}Be concentrations of each of the four laboratories overlap within 1 standard deviation. The average concentration of laboratory 3 does not change. The single result from laboratory 7, also measured on AMS 1, increases by 2.1%. All results reported for these laboratories include this adjustment, for which no additional analytical uncertainties have been included.

To date, 89 ^{10}Be measurements and 23 ^{26}Al measurements have been reported. ^{10}Be and ^{26}Al concentrations are given in Figures 4 and 5, respectively, where each plot shows the results for a single laboratory (the accelerators are given different symbols, laboratories are given different colors). All laboratories had their samples measured on a single AMS facility except laboratories 3 and 10 who measured ^{10}Be at two different accelerators. Blank subtractions reduced ^{10}Be concentrations by between 0.1 to 4.5 %, though the majority of corrections were less than 1% (Figure 6). The larger blank

subtractions are typically associated with smaller sample masses (e.g., <1 g instead of 5 g), rather than higher reagent blank $^{10}Be/^{9}Be$ measurements. All reagent blank subtractions for Al were less than 0.5%. Most laboratories reported blanks that were prepared in the same batch as the CoQtz-N samples, while one laboratory used a long-term average blank value for both ^{26}Al and ^{10}Be. We note that AMS $^{10}Be/^{9}Be$ measurements were normalized to either: the NIST ^{10}Be SRM 4325 assuming the nominal $^{10}Be/^{9}Be$ value of 2.79×10^{-11} (Nishiizumi et al., 2007); the ICN standard dilution series using the values reported in Nishiizumi et al. (2007); or they were calibrated against in-house standards that were traceable to these values. All $^{26}Al/^{27}Al$ AMS measurements were normalized to the standards of Nishiizumi (2004), or to in-house standards, cross-calibrated against the consensus values reported in the round-robin exercise of Merchel and Bremser (2004). Be and Al oxides prepared from CoQtz-N were mixed with either Nb (Be) or Ag (Be and Al) and all of the AMS facilities used standards pressed with the same metal as the CoQtz-N samples.

3.3. Summary statistics for ^{10}Be and ^{26}Al concentrations

We have derived summary statistics for the CoQtz-N results grouped by the laboratory where the samples were prepared. The results reported for each laboratory are not independent of the AMS facility where the measurements were made and this point will be discussed again briefly in section 3.4. For laboratories that obtained more than one result, the arithmetic and error weighted means plus associated uncertainties are shown in Tables 3 and 4. The median may provide a more robust estimate of central tendency when few measurements have been made (Dean and Dixon, 1950) and this, plus the 25^{th} and 75^{th} percentiles, are given for laboratories with five results or more. Figures 7a and 7b compare the arithmetic mean and 95% confidence intervals, plus medians where appropriate, for all laboratories with two or more ^{10}Be or ^{26}Al results. The error weighted mean concentrations and the estimated standard error of the weighted mean for each laboratory are also provided in Tables 3 and 4 but are not

discussed further here. The reduced Chi-square, or mean square weighted deviation (MSWD) values are determined for ^{10}Be measurements from laboratories 1, 2, 3, 6 and 11 (Table 3) and for ^{26}Al measurements from laboratory 1 and 11 (Table 4). MSWD values on the order of 1 may be interpreted to mean that the uncertainties reported for individual measurements from a particular laboratory approximately agree with the scatter observed when a number of replicate measurements are made. In this way, it can be used as an indicator of whether or not laboratories and/or the AMS facilities where the measurements were made, are adequately characterizing their analytical uncertainties. However, the uncertainty in the MSWD value is large when there are few degrees of freedom, that is, when sample populations are small (Wendt and Carl, 1991). Therefore, we do not derive the MSWD when there are less than seven results reported from a given laboratory. The MSWD results are discussed further in section 4.2.

Potential outliers were considered for each individual laboratory. Laboratory 1 has a single, high ^{10}Be measurement that both Grubbs and Dixon methods (Taylor, 1987) identify as an outlier (P=<0.01). This result is identified in Figure 4. However, based on the analytical uncertainties of the individual measurements it is not an unexpected result, as the measurement lies less than three of its standard deviations from the mean laboratory value, where n=21. As neither of the above methods of outlier identification take into account uncertainties and as nothing unusual was observed during the preparation or measurement of this sample that would explain the high value, we do not omit this result. However, we note that it is one of thirteen measurements that are rejected during the second stage of deriving a filtered error weighted mean (see Section 3.4). Neither Grubbs nor Dixon methods identify any outliers in the ^{26}Al results reported.

3.4. ^{10}Be concentration characterization

We compare three different approaches of characterizing the nuclide concentrations of CoQtz-N. The first is the approach of Ku (1969), termed the 'grand average' by Taylor (1987), who suggests its application to pool laboratory averages where different numbers of measurements have been reported and different amounts of dispersion exist. The result is in essence a weighted mean of the laboratory averages, where the weights are inversely proportional to the variances of the mean from each laboratory. This allows laboratories that make more measurements, or that have more tightly clustered datasets to be given more emphasis in the final result. The variance of the grand average is then the reciprocal of the sum of the weights.

The second approach is described by Rozanski et al. (1992) and was used for combining the ^{14}C data from many laboratories during early inter-laboratory comparison exercises (Scott et al., 2003). It is the approach used by Jull et al. (2015) to provide preliminary ^{10}Be concentration values for the Cronus-Earth reference materials. This approach, herein termed the 'filtered EWM' (filtered error weighted mean), considers all the results as a single dataset, rather than taking laboratory averages, and uses two stages of measurement elimination. The first stage rejects measurements more than three multiples of the interquartile range from the median and then calculates a new median. The second stage considers whether individual results that passed stage one deviate from the new median by more than two multiples of their measurement uncertainty, rejecting those that do. The remaining results are used to derive an average value by taking an error weighted mean. This approach aims to remove potential outliers and results whose quoted errors may be underestimated. Additionally, Rozanski et al. (1992) suggest that the Chi-square test proposed by Ward and Wilson (1978) is used to determine whether the remaining results come from a single population, incorporating an additional multiplier to the estimate of the standard error when the null hypothesis is rejected.

The third method we use to characterize the radionuclide concentrations of CoQtz-N is 'linear mixed modelling' and our approach is similar to that of the most recent ^{14}C inter-laboratory comparison exercise (Scott et al. 2017). Mixed (random effect) models allow variance to be partitioned into multiple components, in this case the laboratory and the AMS facility. Unlike the above two methods, it accommodates the interdependency of results that have been prepared in different laboratories but measured using the same AMS facility. For example, it caters for laboratories 1, 2, 3 ,4 and 7 all having measured ^{10}Be using AMS 1 and thus having a mutual source of variance. Our model takes the form shown in equation 2:

$$Y_{ijk} = \mu + \alpha_i + \beta_{ij} + \varepsilon_{ijk}$$

Eq. 2

where Y is the radionuclide concentration measured in the kth sample reported from the jth laboratory and measured at the ith AMS facility. We note that: k = 1,…,K, where K is the number of replicate CoQtz-N measurements from a given laboratory; j = 1,…,J, where J is the number of laboratories; and that i = 1,…,I, where I is the number of AMS facilities. μ is the overall mean value, α is the random effect for an AMS facility, β is the random effect for a preparation laboratory and ε is the deviation from the overall mean not accounted for by the previous two effects.

We employ a hierarchical design that nests laboratories 'within' AMS facilities, as many laboratories may measure at a single AMS facility but the reverse is less common. In the two cases where a single laboratory has measured ^{10}Be in CoQtz-N samples at two different AMS facilities we have treated the results as belonging to two separate groups. For example, for the purposes of the modelling, the results of laboratory 3 are separated and nested within either AMS 1 or AMS 2 (see figure 4).

An estimate of the mean radionuclide concentration and corresponding variance is required from each laboratory in order to calculate a grand average. We include laboratories that have reported three or more measurements (Table 3), so as to get a reasonable estimate of variance, and obtain a result of 2.53 ± 0.02 x 10^6 atoms ^{10}Be/g (estimated 95% Confidence Interval). The filtered EWM calculated according to Rosanski et al. (1992) produces 2.54 ± 0.02 x 10^6 atoms ^{10}Be/g (estimated 95% Confidence Interval). During derivation of filtered EWM no measurements were rejected in stage 1. Thirteen of the eighty-nine measurements were rejected during stage 2 (Figure 8). We fail to reject the null hypothesis that the remaining dataset is homogeneous at the 5% level and so do not add a multiplier to the estimate of the standard error of the filtered EWM. The linear mixed modeling utilizes all the data and estimates a mean value of 2.53 ± 0.09 x 10^6 atoms ^{10}Be/g (95% Confidence Interval). The above results are summarized in Table 5.

3.5. ^{26}Al concentration characterization

Fewer measurements of ^{26}Al are reported than are of ^{10}Be. Nevertheless, we derive a grand average and consensus value as per ^{10}Be. For the grand average we calculate a value of 15.2 ± 0.1 x 10^6 atoms ^{26}Al/g (estimated 95% Confidence Interval). The result is clearly dominated by laboratory 6, whose three measurements are very tightly clustered and thus strongly biasing the error weighted result. The filtered EWM calculated according to Rosanski et al. (1992) gives 15.7 ± 0.4 x 10^6 atoms ^{26}Al/g (estimated 95% Confidence Interval). During derivation of the filtered EWM, no measurements were rejected in stage 1 and four of twenty-three values were rejected in stage 2 (Figure 9). There is no evidence to reject the null hypothesis that the dataset is homogeneous at the 5% level and so no error multiplier is included in the estimate of the standard error of the weighted mean. The result of our linear mixed modeling is a mean value of 15.6 ± 1.6 x 10^6 atoms ^{26}Al/g (95% Confidence Interval). The above results are summarized in Table 5.

4. Discussion

4.1 Material purity

There is no difference observed in ^{10}Be concentrations of CoQtz-N after additional HF/HNO_3 etches (Figure 2). This suggests that the cleaning of the CoQtz-N material, performed prior to splitting, was sufficient to remove any adsorbed meteoric ^{10}Be. Further cleaning of CoQtz-N is unnecessary and to reduce potential sources of contamination it should not be further processed, chemically or otherwise, prior to dissolution.

The low scatter of the five ICP-OES Al concentration measurements suggest the Al concentration within CoQtz-N splits is uniform (Table 2). Na, K, Ti and Fe show greater variability and this scatter appears largely due to one of the five 1 g portions having higher concentrations of these particular elements. This might reflect the presence of minor amounts of non-quartz minerals (e.g., rutile in the case of Ti). In any case, the concentrations of these elements are also low.

4.2 Intra-laboratory results of CoQtz-N measurements

The MSWD of the ^{10}Be results from laboratories 1 and 3 are somewhat lower than unity, at 0.49 and 0.52 respectively. The probability of obtaining an MSWD result equal to or lower than that of laboratory 1 is ~3%, meaning there is a reasonably strong likelihood that this laboratory overestimates the size of its measurement uncertainties. The MSWD result from laboratory 3 is similar to laboratory 1 but because of fewer results (i.e. fewer degrees of freedom) it is less statistically significant, with a ~16% probability of obtaining an equal or lower MSWD value. This means it is premature to consider whether laboratory 3 is also overestimating uncertainties. The ^{10}Be concentrations from laboratory 6 result in an MSWD value of 3.46 and the probability of such a result occurring by chance is <1%. This strongly

suggests that laboratory 6 is underestimating analytical uncertainties. As the majority of laboratories do not show excess scatter (Table 3), the individual splits of CoQtz-N seem to be internally homogeneous with regard to the ^{10}Be concentration, at least within typical measuring precisions of a few percent. Thus, bias should not be introduced during the sub-sampling of the splits and the material can be spooned directly from the beakers without the need for additional splitting methods. The MSWD value for ^{26}Al measurements from laboratory 1 is close to unity but the result from laboratory 11 is high at 2.32, with a probability of obtaining such a result, or higher, being ~3%. These laboratories MSWD values are constrained by a relatively small number of results (Table 4), thus, we suggest that there is within-split homogeneity also for ^{26}Al but we note that this assertion is tentative pending more data.

Laboratories 3 and 10 measured ^{10}Be on 2 different AMS (Figure 4). Laboratory 3 measurements from AMS 1 (n=4) and AMS 2 (n=5) show good agreement, suggesting these accelerators produce comparable results. The small number of measurements from laboratory 10 makes it premature to compare results. More comparisons between single laboratories and multiple accelerators are required.

4.3 Inter-laboratory results of CoQtz-N measurements

The stable ion currents of ^{10}Be/^{9}Be measurements, relative to the output of the standards, are seen as a general indicator of the quality or performance of the target. The large number of CoQtz-N ^{10}Be/^{9}Be measurements made by AMS 1 from targets prepared by five different laboratories shows that ^{10}Be concentrations may vary in relation to target performance as seen in Figure 3. It is not clear that this co-dependency is entirely attributable to the specific configuration of AMS 1 (i.e. tuning, transmission, ion source behavior, etc.) but normalizing the data to correct for this effect resolves what would otherwise be a discrepancy between average CoQtz-N ^{10}Be concentrations. The chemistries employed by laboratories 1 through 4 and laboratory 7 for target preparation are different and will likely have a

fundamental control over the typical relative stable ion currents. Several other factors could also contribute to the average laboratory target performance, including: 1. the experience and skill of those preparing the targets; 2. the typical research applications of the research group, which can dictate the purity of the starting material, i.e. fluvial or colluvial sediments can be relatively rich in resistant mineral impurities, making it more challenging to obtain pure quartz and subsequently to separate Be from other elements during sample processing (Binnie et al., 2015; Corbett et al., 2016; Mifsud et al., 2013); 3. the type of metal matrix used during target pressing, the ratio of metal to BeO and the homogeneity of the mixing between the metal and the oxide are important considerations (Hunt et al. 2006; Merchel et al., 2008; Stone et al., 2004). The range of ^{9}Be currents that were obtained by AMS 1 during the measurements of CoQtz-N from multiple laboratories allows the recognition of a relationship with ^{10}Be concentrations and current that might not be apparent if the target measurements had performed within a narrower range of currents, as is often the case for measurements of an in-house standard from individual target preparation laboratories. More generally, findings such as this illustrate the advantage of different laboratories and accelerators making many measurements of quartz reference materials, such as CoQtz-N, over the long-term.

The usefulness of CoQtz-N to the user community depends in part on an appropriate characterization of the nuclide concentrations it contains. Here we considered three approaches for this determination, termed the grand average, the filtered EWM and linear mixed modelling. The near identical ^{10}Be concentrations derived by the different methods may be coincidental but provide some confidence in the result. For the grand average ^{10}Be concentration we included laboratories with three or more measurements, but including only the three laboratories with the most measurements (laboratories 1, 2 and 11, n≥10) would increase the average by only 0.4%. This is because weighting by the inverse of the variance of the mean (n/σ^2) has the consequence that the grand average is strongly influenced by those

laboratories that have reported the most measurements. In contrast, the difference found between the averaging methods when it comes to ^{26}Al concentrations is due to the very low spread (i.e. excellent reproducibility) of the three values from laboratory 6 and the tendency of the grand average to place more weighting on mean values with low dispersion. Thus, in the case of ^{26}Al there is a strong bias towards the mean of laboratory 6 that does not occur in the filtered EWM or linear mixed modelling approaches, which treat every measurement separately. In general, ^{26}Al concentration measurements tend to have higher analytical uncertainties and to show more inter-laboratory scatter than ^{10}Be (Jull et al., 2015; Phillips et al., 2016) and so our characterization of the ^{26}Al concentration of CoQtz-N would benefit from more measurements. There is little difference between the average values estimated using the filtered EWM and linear mixed modelling approaches, but linear mixed modelling recognizes there are common sources of variation and provides appropriately adjusted uncertainties. Accordingly, we favor the results of the linear mixed modelling as preliminary concentrations for both ^{10}Be and ^{26}Al.

By standardizing methods of concentration calculation we aimed to exclude blank subtraction as an additional source of dispersion between the CoQtz-N results. Other causes for the differences observed could include inappropriate characterization of ^{9}Be carrier concentrations (Merchel et al., 2013), or incorrect determination of the ^{27}Al in the sample (Fujioka et al., 2015). Precise and accurate determination of ^{27}Al is as critical as the quality of the ^{26}Al AMS measurement. In addition, improper calibration of laboratory balances, or non-trivial effects of static during weighing, can be problematic, as can systematic bias introduced by individuals preparing the targets. Indeed, this last effect in particular is difficult to investigate unless there is already a long-term record of laboratory in-house standards. In addition, as discussed above, comprehensive examinations of target performance effects are needed. It is not yet possible to resolve whether the differences we observe between averages calculated at the level of individual laboratories are introduced during the processing or during the AMS measurements.

Though the differences between the average ^{10}Be concentrations from various groups are small, they are more apparent in the few ^{26}Al results. Greater distribution of CoQtz-N targets from individual laboratories to several AMS facilities would help address these issues and such tests will be implemented in the near future.

5. Conclusions

Initial ^{10}Be and ^{26}Al results of CoQtz-N show that it is suitable as an in-house reference material and for inter-laboratory comparisons. Based on the available results, consensus values for ^{10}Be and ^{26}Al in CoQtz-N are 2.53 ± 0.09 x10^6 at/g and 15.6 ± 1.6 x10^6 at/g, respectively, at the 95% confidence limit. These values should be considered preliminary and the ^{26}Al value in particular is likely to be subject to changes in the future as more results are forthcoming. It is encouraging that there appears a reasonable agreement in ^{10}Be concentrations prepared in different laboratories and measured on several AMS. ^{26}Al concentration measurements show less concordance and clearly require focused attention in future. It is not yet clear whether the differences we observe stem from the target preparation or the AMS measurement. More measurements from a single laboratory at multiple AMS facilities would help address this and ongoing determinations will help to improve our understanding of what is causing dispersion. To obtain a split of CoQtz-N contact S. Binnie or T. Dunai.

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Figure captions

Figure 1. The starting material for CoQtz-N was the single boulder of vein quartz indicated.

Figure 2. ^{10}Be measurements of CoQtz-N that have undergone further HF/HNO_3 etching in addition to that undertaken during bulk preparation. The ^{10}Be concentrations are unaffected by more etches, suggesting the material is free of any adsorbed meteoric ^{10}Be.

Figure 3a. ^{10}Be measurements of CoQtz-N from AMS 1 display a positive relationship with the analytical ^{9}Be current of the individual targets, represented as a fraction of the average current of the standards in the same run (relative ^{9}Be current). A linear relationship is indicated (r = 0.43) and the grey lines are the 95% confidence intervals. **3b** shows the ^{10}Be concentrations of the same samples as in figure 3a following normalization to the average standard currents from the same run.

Figure 4. CoQtz-N ^{10}Be measurements from each laboratory where the targets were prepared. Each laboratory is given a different colored symbol and labelled as Lab 1 through Lab 11. The shape of the symbols refers to the accelerator mass spectrometers where the targets were measured, labelled AMS 1 through AMS 7. Error bars are one standard deviation, the thick grey line is the arithmetic mean of all measurements and the thinner grey lines are the 95% confidence intervals of the mean. The asterisk (*) denotes a potential outlier from laboratory 1 (see main text for more details).

Figure 5. CoQtz-N ^{26}Al measurements from each laboratory where the targets were prepared. Error bars are one standard deviation, the thick grey line is the arithmetic mean of all measurements and the thinner grey lines are the 95% confidence intervals of the mean.

Figure 6. The upper plot shows all ^{10}Be concentrations with the same symbol color and shape scheme as in Figure 4. The lower plot shows the corresponding reagent blank subtractions for each individual measurement.

Figure 7a. Arithmetic mean ^{10}Be values for each target preparation laboratory where two or more results were obtained and the corresponding 95% confidence intervals where there are 3 or more results (see Table 3). The median ^{10}Be concentration, indicated by the red squares, is also derived for laboratories with three or more results. **7b**. as in **7a** but for ^{26}Al results. See Table 4 for details.

Figure 8. Ranked ^{10}Be concentrations measurements of CoQtz-N, including one standard deviation uncertainties on the individual measurement. The 13 red circles are the measurements that are rejected prior to the derivation of the error weighted mean, according to Rozanski et al. (1992). The error weighted mean ^{10}Be concentration is given by the black line and the estimated 95% confidence intervals of the weighted mean are illustrated by the green lines. Note the confidence intervals are derived from the estimated standard error of the weighted mean.

Figure 9. Ranked ^{26}Al concentrations measurements of CoQtz-N including one standard deviation uncertainties on the individual measurements. The four red circles are the measurements that are rejected prior to the derivation of the error weighted mean, according to Rozanski et al. (1992). The error weighted mean ^{26}Al concentration is given by the black line and the estimated 95% confidence intervals of the weighted mean are illustrated by the green lines. Note the confidence intervals are derived from the estimated standard error of the weighted mean.

Figure 1 (single column width). Online version (greyscale copy to be provided for print version).

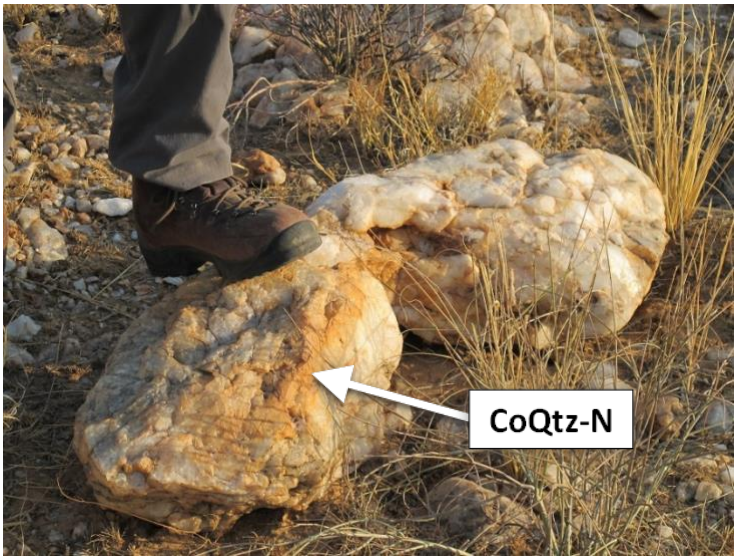


Figure 2 (single column width).

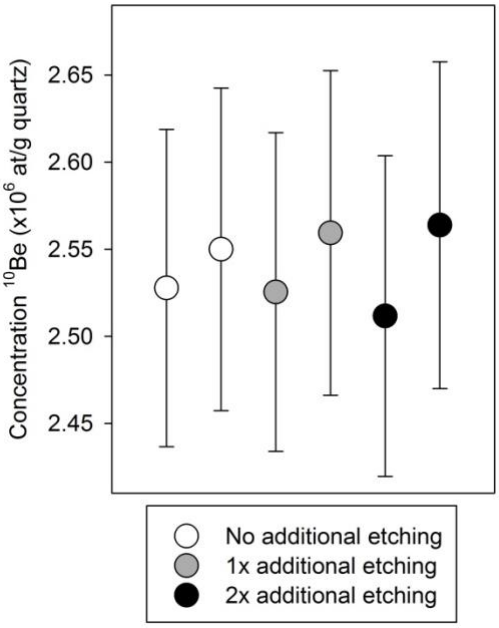
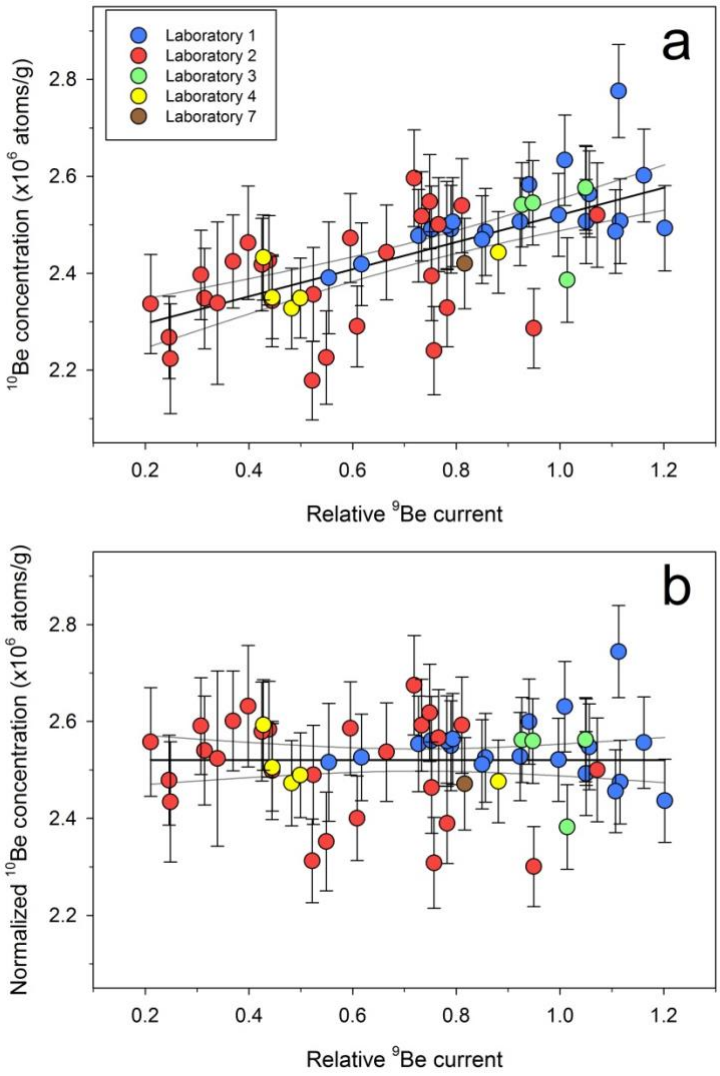


Figure 3 (single column width). Online version (greyscale copy to be provided for print version).



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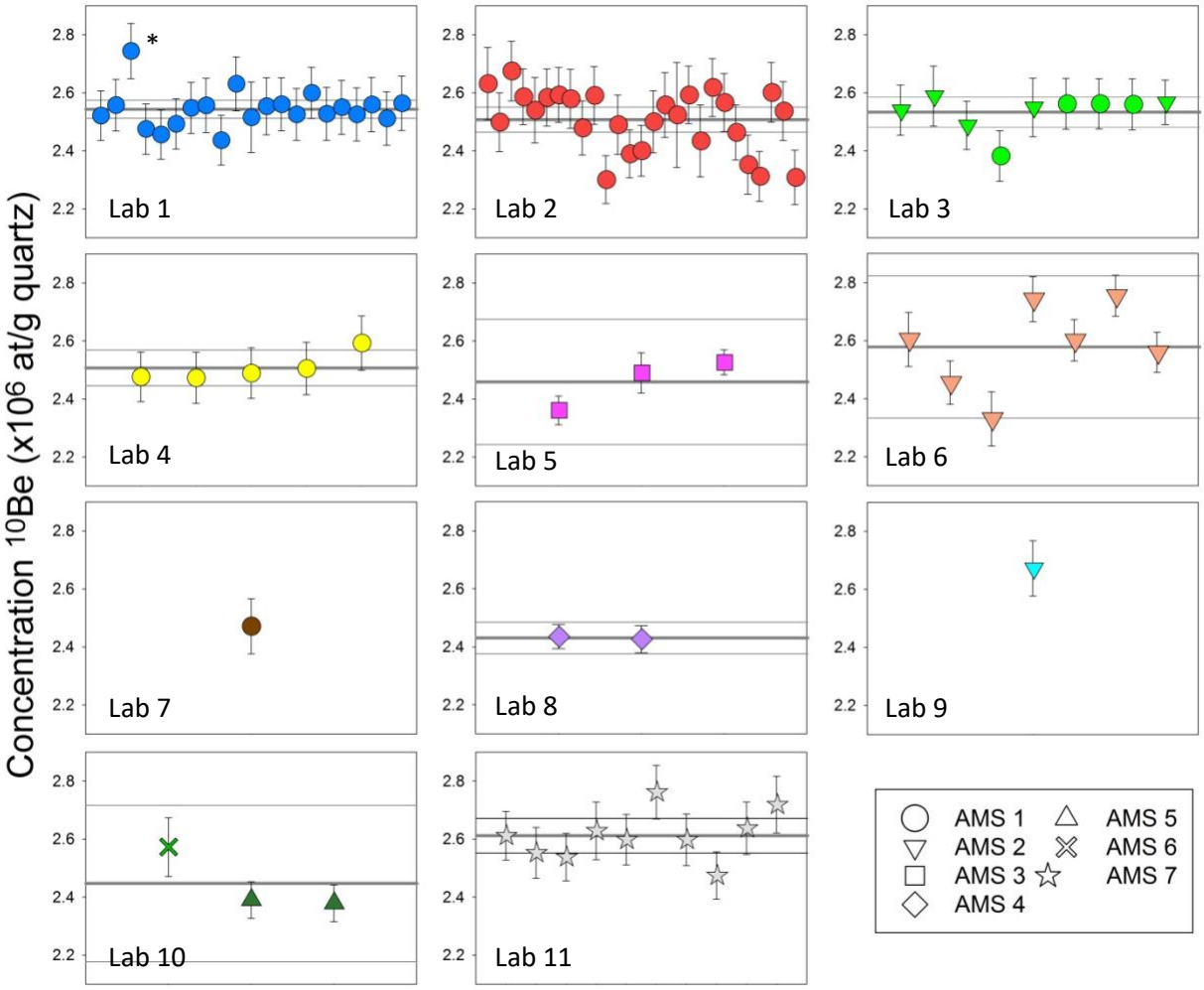
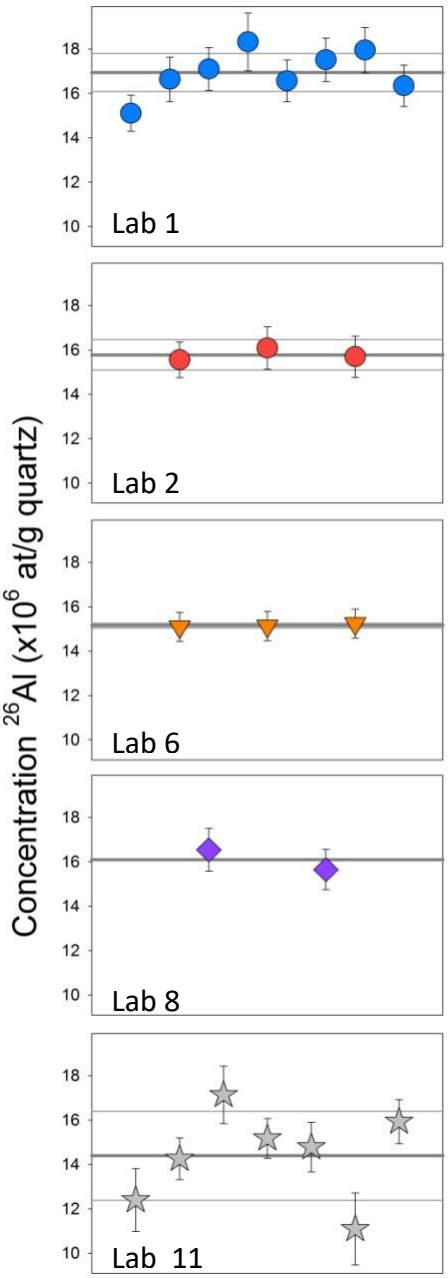


Figure 5 (single column width). Online version (greyscale copy to be provided for print version).



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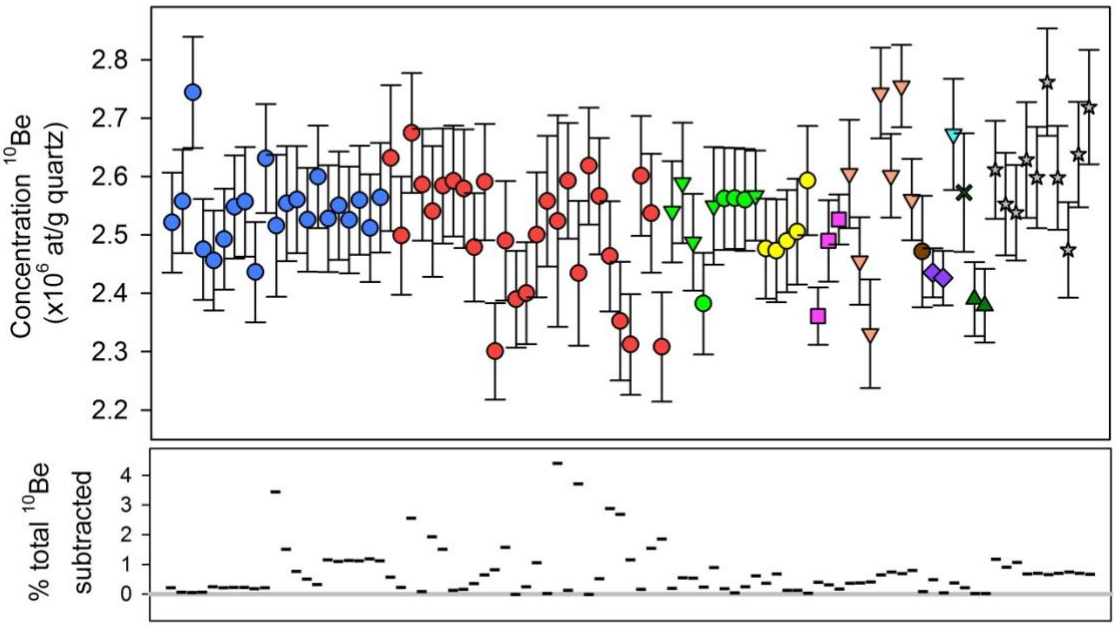

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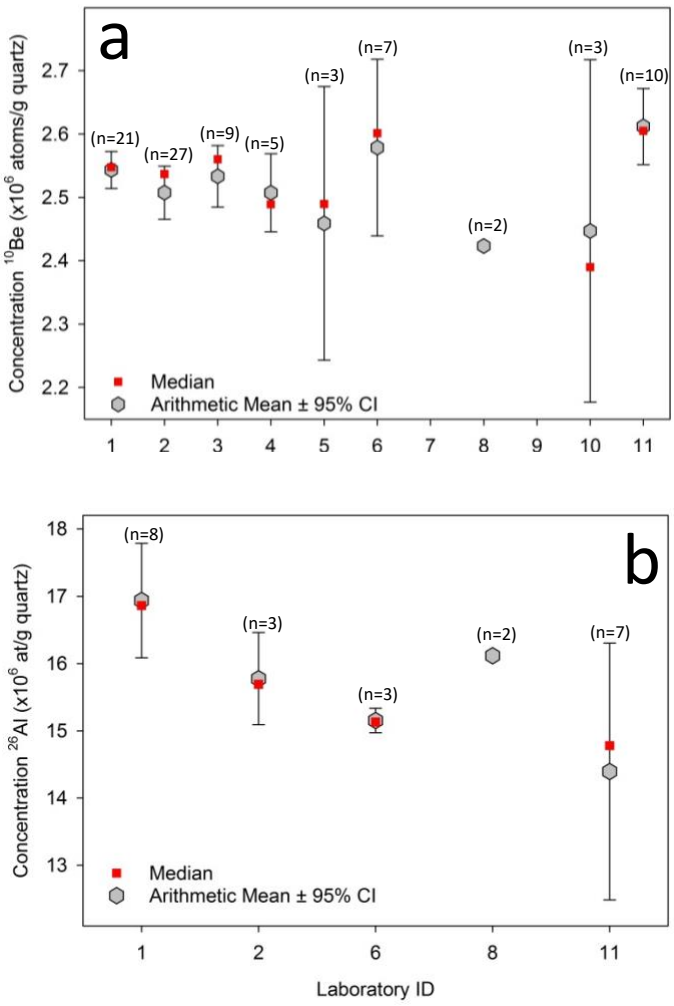

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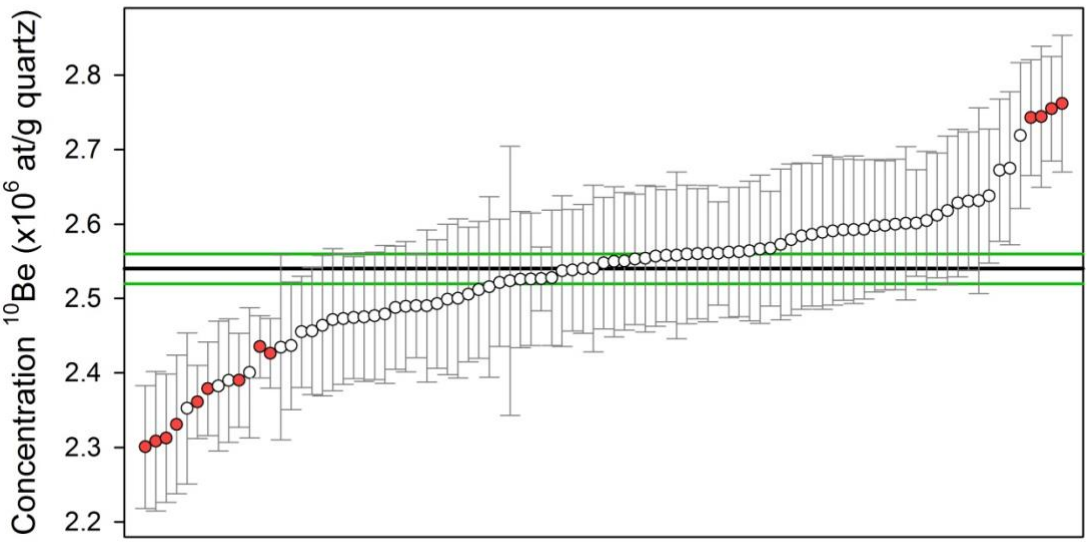

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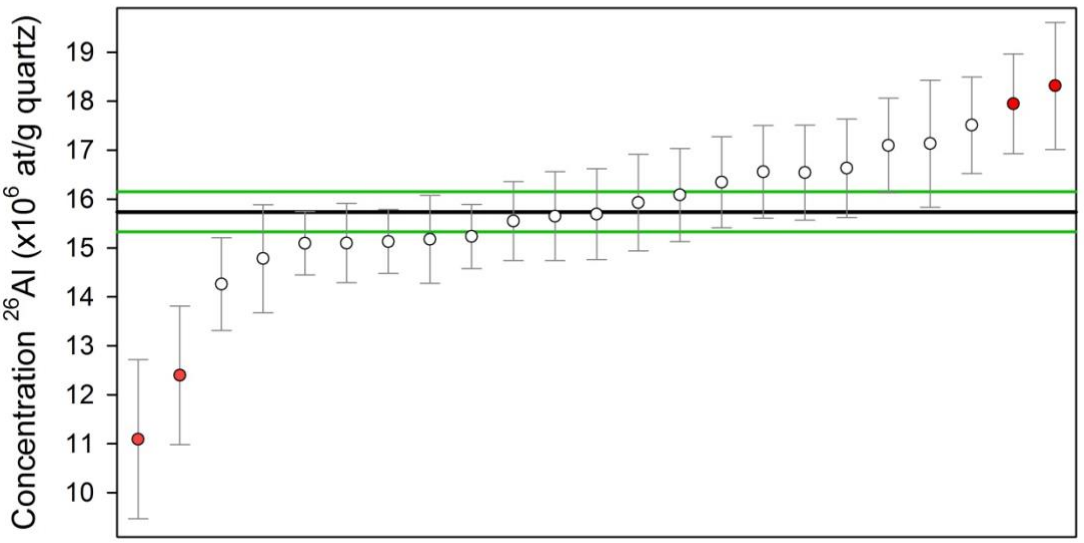

Table 1. (Single column width)

Table 1.
Participating laboratories and AMS facilities (alphabetical order)

Target Preparation Laboratories

- Australian Nuclear Science and Technology Organisation (ANSTO)
- Centre Européen de Recherche et d'Enseignement des Géosciences de l'Environnement (CEREGE)
- Eidgenössische Technische Hochschule (ETH-Zurich)
- GeoForschungsZentrum (GFZ)
- Scottish Universities Environmental Research Centre (SUERC)
- University of Bremen
- University of Bern
- University of Cologne
- University of Münster
- University of Tübingen
- University of Wollongong

Participating AMS Facilities

- Accélérateur pour les Sciences de la Terre, Environnement, Risques (ASTER)
- Australian National University (ANU)
- Australian Nuclear Science and Technology Organisation (ANSTO)*
- Eidgenössische Technische Hochschule (ETH-Zurich)
- Scottish Universities Environmental Research Centre (SUERC)
- University of Cologne (CologneAMS)

*Measurements made on two AMS, Antares and Sirius.

Table 2 (Single column width)

Table 2: CoQtz-N major elements by ICP-OES

Element	CoQtz-N concentration[1]
Na	7.7 ± 1.9
Al	18.4 ± 0.6
K	2.3 ± 1.6
Ca	1.3 ± 0.1
Ti	7.6 ± 2.6
Fe	16.6 ± 14.5
All of Be, B, Mg and Mn are sub-ppm	

[1] Mean and 1 standard deviation (n=5). Measured in-house at University of Cologne on a Spectro Arcos ICP-OES, using standard addition (four dilutions), wavelengths (nm): Be 313.107; B 249.773; Na 589.592; Mg 280.270; Al 167.078; K 766.491; Ca 396.847; Ti 334.941; Mn 257.611; Fe 261.187.

Table 3 (Double column width)

Table 3.
Different measures of ^{10}Be concentration central tendency. s.d. and s.e. refer to the standard deviation and standard error, respectively. HL and HU refer to the lower and upper quartiles (hinges). The mean square weighted deviation (MSWD) is included for laboratories that have reported seven or more measurements.

Target Prep. Lab.	AMS fac.	N	Arithmetic Mean	±1 s.d.	±1 s.e.	95% Confidence Interval [LL - UL]	Error Weighted Mean (EWM)	Estimated ±1 s.e. of the EWM	Median	Quartiles [H_L – H_U]	MSWD
			(x10^6 at/g quartz)								
1	1	21	2.54	0.06	0.01	2.51 - 2.57	2.54	0.02	2.55	2.52 - 2.56	0.49
2	1	27	2.51	0.11	0.02	2.47 - 2.55	2.50	0.02	2.54	2.45 - 2.59	1.27
3§	1,2	9	2.53	0.06	0.02	2.48 - 2.58	2.53	0.03	2.56	2.54 - 2.56	0.52
4	1	5	2.51	0.05	0.02	2.45 - 2.57	2.50	0.04	2.49	2.48 - 2.51	.
5	3	3	2.46	0.09	0.05	2.24 - 2.67	2.46	0.03	2.49	.	.
6	2	7	2.58	0.15	0.06	2.44 - 2.72	2.59	0.03	2.60	2.51 - 2.67	3.46
7*	1	1	2.47	0.10	.	.	.	.	.	.	.
8	4	2	2.43	0.00	0.00	.	2.43	0.03	.	.	.
9*	2	1	2.67	0.10	.	.	.	.	.	.	.
10§	5, 6	3	2.45	0.11	0.06	2.18 - 2.72	2.41	0.04	2.39	.	.
11	7	10	2.61	0.08	0.03	2.55 - 2.67	2.60	0.03	2.60	2.56 2.64	0.90

* A single measurement is reported by these laboratories, the single value and quoted uncertainty are shown under arithmetic mean and standard deviation.
§ Where two AMS facilities have been used the statistics are derived using the combined measurements from both.

Table 4 (Double column width)

Table 4.
Different measures of ^{26}Al concentration central tendency. s.d. and s.e. refer to the standard deviation and standard error, respectively. HL and HU refer to the lower and upper quartiles (hinges). The mean square weighted deviation (MSWD) is included for laboratories that have reported seven or more measurements.

Target Prep. Lab.	AMS Fac.	n	Arithmetic Mean	±1 s.d.	±1 s.e.	95% Confidence Interval [LL - UL]	Error Weighted Mean (EWM)	Estimated ±1 s.e. of the EWM	Median	Quartiles [HL – HU]	MSWD
			($x10^6$ at/g quartz)								
1	1	8	16.9	1.0	0.4	16.1 – 17.8	16.7	0.3	16.9	16.5 - 17.6	1.18
2	1	3	15.8	0.3	0.2	15.1 – 16.5	15.7	0.5	15.7	.	.
6	2	3	15.2	0.1	0.0	15.0 – 15.3	15.2	0.4	15.1	.	.
8	4	2	16.1	0.6	0.4	.	16.1	0.7	.	.	.
11	7	7	14.4	2.1	0.8	12.5 – 16.3	14.8	0.4	14.8	13.3 - 15.6	2.32

Table 5 (Single column width)

Table 5.
The average values and confidence intervals for concentrations of ^{10}Be and ^{26}Al in CoQtz-N derived using the three different approaches described in detail in the main text.

Averaging approach used	Average ^{10}Be concentration ± 95% Confidence Interval (x10^6 at ^{10}Be /g)	Average ^{26}Al concentration ± 95% Confidence Interval (x10^6 at ^{26}Al /g)
Grand Average	2.53 ± 0.02	15.2 ± 0.1
Filtered EWM	2.54 ± 0.02	15.7 ± 0.4
Linear Mixed Modeling	2.53 ± 0.09	15.6 ± 1.6